Mr. Lovingood

DPM
3128

TO PREPARE TEFLON® FOR BONDING

TETRA-ETCH®

GORE

W. L. GORE & ASSOCIATES, INC.
Newark, Delaware / Flagstaff, Arizona

Cementing or potting Teflon* used to be a problem. But that was before the invention of TETRA-ETCH. Either Teflon was not cemented at all, or a hazardous, difficult process was used. Now Teflon and other fluorocarbon polymers can be easily and safely etched for potting and bonding using W. L. Gore & Associates' TETRA-ETCH.

WHAT IS IT?

TETRA-ETCH is a non-pyrophoric chemical solution for treating the surface of Teflon to make it markable, bondable and pottable. A sodium compound in the solution reacts with highly fluorinated polymers to form a reactive film on the polymer surface. Just about any adhesive can be used on this treated surface.

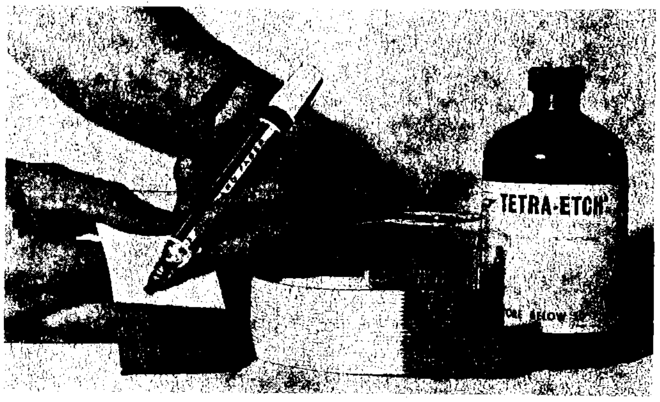
HOW DOES IT WORK?

The active sodium in TETRA-ETCH reacts with the surface molecules of a halogenated polymer to form a carbonaceous film of free radicals. These radicals react with O_2 and H_2O and each other to form a layer of chemical groups attached to the surface. Typical groups formed are: ethylenic, acetylenic, hydroxyl and carbonyl. Many of these react with adhesives and potting compounds to form true chemical bonds.

HOW IS IT USED?

The surface must be dry and clean. The object to be treated can be dipped into the solution. Or TETRA-ETCH can be spread on the surface with a strip of metal or polyethylene, neither of which react with TETRA-ETCH. TETRA-ETCH is dark green as it is applied. When the reaction is completed the treated surface has a brownish cast. Bondability is indicated by the change of color and not by the hue or darkness. Another test: a marking pen will easily mark the etched surface. After treatment, the surface must be thoroughly washed to remove all residue. Part of the residue is soluble in water and part in organic solvents only. Washing the surface first in methanol and then in water is one simple way to clean the surface. An alternate way is washing in water to remove some of the water soluble residue; washing in naptha gasoline or any of the chlorinated solvents to remove the residue insoluble in water; then again with water to remove water soluble residue trapped by the insoluble residue.

* DuPont trademark



WHAT HAPPENS IF THE SURFACE IS UNWASHED?

Residue left on the treated surface reacts with moisture in the air to form caustic soda, which in time attacks the etch. The etch can then be wiped away, once again leaving a Teflon surface unsuitable for bonding.

HOW STRONG IS THE BOND?

Peel strengths of bonds range from 5 to 35 lbs. per inch, depending on the type of adhesive, the thickness of the adhesive and the surfaces being joined.

Shear strengths generally range from 60 to 130 lbs. per inch.

HOW ABOUT POTTING?

Exhaustive tests on Teflon cables etched with TETRA-ETCH and potted in connectors show no leakage. The cables were immersed in water repeatedly, twisted, bent, pulled and immersed in borax and water, but the bond remained intact between the Teflon and the potting compound.

WHAT KIND OF ADHESIVES CAN BE USED?

Just about any adhesive, or potting compound can be used on a surface treated with TETRA-ETCH. For Teflon treated with TETRA-ETCH, for maximum bond strength and resistance to vibration, use an epoxy adhesive such as C. H. Biggs', Helix Bonding AGENT R-363, Emerson & Comings', ECCOBOND 45 or Rubber and Asbestos Corporation's G-379. A pressure sensitive adhesive, satisfactory up to 390°F, is Dow Corning's C271 silicone.

ARE THERE ANY ADVANTAGES IN MARKING?

... standard marking system can be used to mark a surface prepared with TETRA-ETCH. Special inks can be eliminated and marking speeds drastically increased using Teflon cable etched with TETRA-ETCH. Marking can be made clear and legible, assuring quick and accurate identification of leads.

HOW FAST IS IT?

Only a few seconds are required to etch TFE Teflon. Precise timing is unnecessary since the reaction is self-limiting. The carbonaceous film formed in etching retards further etching. Prolonged exposure (minutes) will deepen the etch, but will not improve bond strength.

FEP Teflon, Kel-F* and Kynar† take a bit longer. The reaction may be speeded up and the etch darkened on these materials by heating them to 100°C before etching.

CAN MATERIAL BE OVER-ETCHED?

If the Teflon is left exposed to the etchant excessively long (several hours), a darker brown, slightly thicker film will form. This thick layer is not as strong as a thin one, and is more likely to be separated from the base polymer.

HOW ABOUT A PRODUCTION LINE?

Figure 1 illustrates a continuous line etching method. TETRA-ETCH is placed in an appropriate container and a layer of naptha is floated on it. (TETRA-ETCH reacts with the moisture in the air, decreasing in strength with time.) The cable or film is run through the solution, through a methanol bath, and then a water bath.

A non-flammable system may be constructed by sweeping the TETRA-ETCH with nitrogen. The cable is then washed in a water bath, a chlorinated solvent, and a water bath in turn. Etching rates of 50 to 100 feet per minute are common.

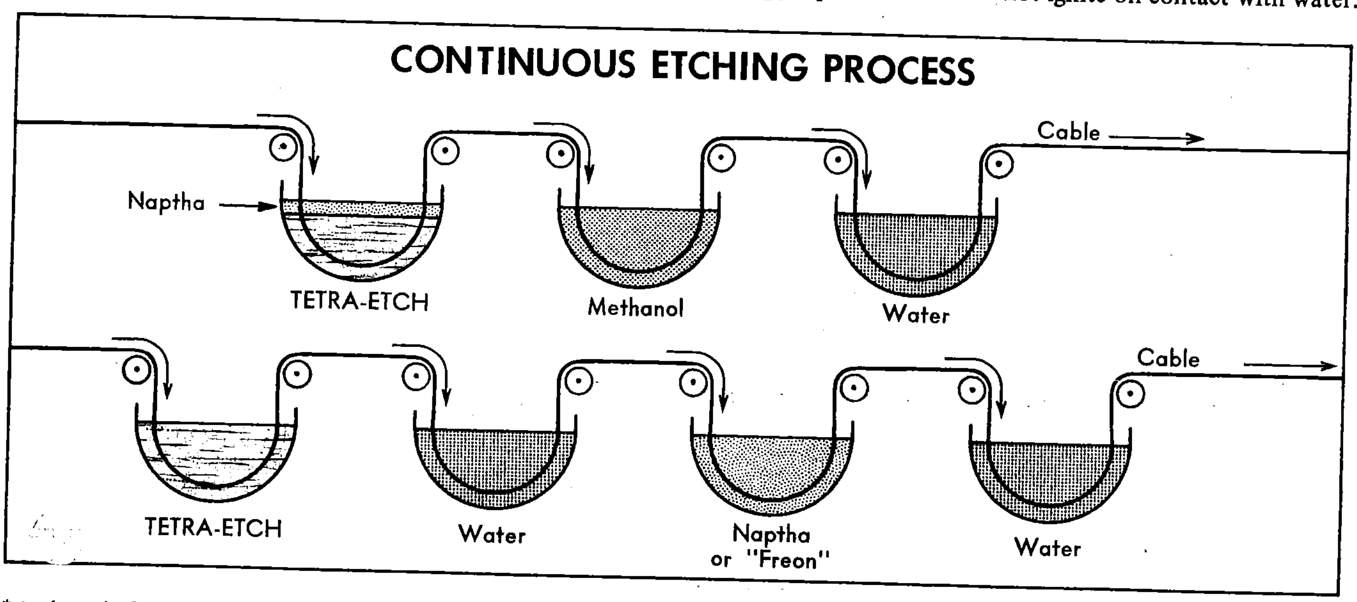
IS CABLE AVAILABLE ALREADY ETCHED?

Gore Associates is equipped to do spot, batch, and continuous etching on a job-lot or production basis. Teflon insulated wire and ribbon cable can be etched in Gore's normal manufacturing process. Etched wire and cable can be furnished at little additional cost.

IS IT FLAMMABLE?

TETRA-ETCH has an exclusive and important property eliminating a major fire hazard—it does not have any free metallic sodium present in it. It will not ignite when exposed to air or poured into water. It is flammable, however, when exposed to open flame. Its properties are shown in the table below.

Color	Dark Green
Specific Gravity	Approximately .97
Boiling Point	Approximately 85°C
Flash Point	33°F
Auto Ignition Point	> 745°C
Solids Content	Approximately 32%
Titration	(Butyl Alcohol))0.5 gram equivalents of active sodium compound per liter. End point is disappearance of dark color.
Non-pyrophoric	Will not ignite on contact with water.

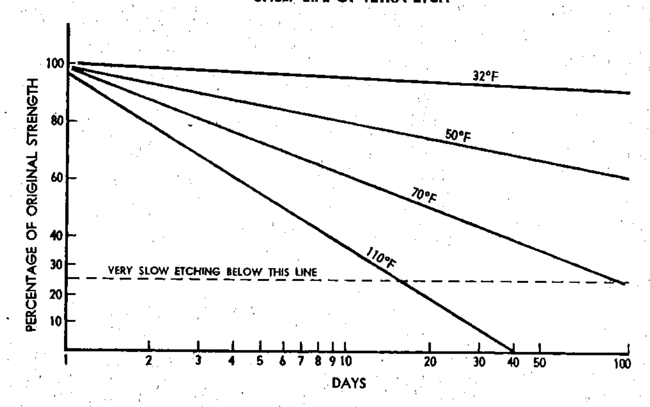


* trademark, 3-M Co.
† trademark, Pennsalt Chemicals Corp.

WHAT SAFETY PRECAUTIONS MUST BE TAKEN?

TETRA-ETCH is flammable and must be kept away from open flame. When the sodium aryl compound in TETRA-ETCH comes in contact with moisture, caustic soda is formed. Solution that comes in contact with the skin should be flushed off with water. Safety glasses should be worn. Protective gloves may be worn. It is harmful if taken internally, chiefly because of the caustic soda that is formed. The vapors from the solvents in TETRA-ETCH are rated non-toxic with reasonable ventilation.

SHELF LIFE OF TETRA-ETCH



HOW ABOUT DISPOSAL?

Disposal is no problem with TETRA-ETCH. Since most of the componets are water soluble, spent or waste TETRA-ETCH can be simply poured down the drain.

HOW LONG DOES IT LAST?

The strength of TETRA-ETCH decreases with exposure to the atmosphere. The sodium in the solution reacts with moisture in the air to form sodium hydroxide. Heat also degrades the solution, as shown on the chart. A two-ounce bottle can be left open to the air for over an hour without appreciable loss of etching ability, but for prolonged exposure in a vat or tank, a layer of naptha on the surface will prevent premature depletion.

TETRA-ETCH is shipped in polyethylene bottles, hermetically sealed in tin cans. Shelf life is guaranteed six months when stored below 50°F. No refrigeration is required for solution used immediately (within a week).

HOW LONG DOES THE ETCHED SURFACE LAST?

Samples stored several years indoors showed no decrease in bondability. The etched surface is affected by ultra-violet radiation, however, and the etch will deteriorate when exposed to sunlight.

WHO SELLS IT?

TETRA-ETCH is manufactured by W. L. Gore & Associates, Inc., Newark, Delaware. TETRA-ETCH orders are filled and shipped from fresh stock, usually on the same day as the order is received. Air shipments are recommended to insure maximum strength.

GORE
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DOUGLAS

MISSILE & SPACE SYSTEMS DIVISIC
DOUGLAS AIRCRAFT COMPANY, INC.

Catalog No. PDL 43538

7-88 (REV. 1 62)

MATERIAL & PROCESS ENGINEERING LABORATORY REPORT

SERIAL NO. MP 14,295

DATE August 17, 1964

TITLE BONDIZING TEFLON TFE AND FEP INSULATED WIRE

ASSIGNED TO C. T. McMurray, A-260

REQUESTED BY D. J. Angeloni, A-260

COPIES TO D. Abelar, A2-260
T. J. King, A-260
H. K. Lauer, A-260
E. Leard, A3-260
R. Q. Morris, A-260
J. M. Okada, A3-260
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H. G. Schlundt, A-260
R. A. Siebert, A-260
File

MATERIALS:

See Materials Index

OBJECT:

To investigate bondizing Teflon insulated wire for adhesion of electrical potting compounds.

PROCEDURE:

A. The Effect of Different Bondizing Formulations

1. Thirty-five TFE (polytetrafluoroethylene) insulated wires (OD 0.060") and thirty-five FEP (fluorinated ethylene procylene) insulated wires (OD 0.113") were cut to six inch lengths and cleaned with a cloth dampened with MEK (methyl ethyl ketone).

2. Five TFE and five FEP wires were immersed in each solution for 30 seconds. "Bond aid", "Tetra-Etch", three month old production solution (DPS 3.114) and the four solutions listed at the end of this procedure were utilized.

3. Within five minutes after removal from the bondizing solutions, the wires were wiped with a clean cloth dampened with MEK.

4. The bondized wires were potted to a depth of one inch in cylindrical silicone molds one inch in diameter (see figure 1). Using Hysol 2039 mixed with Hysol 3561 hardener. The potting compound was cured for 72 hours at 77 ± 5°F and 50 ± 5% RH.

5. The wires were pulled from the potting compound at a rate of 100 lbs per minute at 77 ± 5°F and the load recorded. The shear strength was calculated using the following formula:

$$\text{Shear Strength} = \frac{\text{load (in lbs)}}{\pi \times D \text{ (in inches)} \times \text{length (in inches)}}$$

where D was the mean diameter of the wire insulation before pulling. The results are in table I.

PROCEDURE: (CONTINUED)

B. Coated Wires (Non Bondized)

1. Five wires of each type were cut and cleaned as in paragraph A. 1. The wires were coated with Hitemps' Polymer Coat and Adbond. The coated wires were potted and tested per paragraphs A. 4. and A. 5. The results are recorded in table 2.

2. Five Surbond Type E wires (diameter 0.062") and five Surbond Type EE wires (diameter 0.060") from Surprenant Mfg. Co. were cleaned per paragraph A. 1. They were potted and tested per paragraphs A. 4. and A. 5. The results are in table 2.

C. Effects of Varying Bondizing Time

1. Four sets of insulated wires each set consisting of five of each type (TFE and FEP) were cut and cleaned per paragraph A. 1.

2. Using formula a, the wires were bondized by immersion for 2, 10, 30 and 120 seconds. After cleaning per paragraph A. 3., the wires were potted and tested per paragraphs A. 4. and A. 5. The results are in table 3.

D. Effect of Ultraviolet Light on the Bondized Surface

1. Four sets of five TFE wires were cut and cleaned per paragraph A. 1.

2. The wires were bondized for 30 seconds in formula a and cleaned with MEK.

3. The first set was kept in total darkness. The second set wrapped with two layers of red cellophane (as a shield against ultraviolet light) and placed in the weatherometer two feet from the carbon arc. The third set was placed next to the cellophane wrapped wires in the weatherometer. The fourth set was placed one foot from an unfiltered, 100 watt Westinghouse CH-4 projector spot lamp. The three sets of wires were exposed to the ultraviolet sources for 16 hours.

4. The wires were then cleaned with MEK and potted per paragraph A. 4. They were tested per paragraph A. 5. The results are in table 4.

The following formulations were investigated:

a. Standard formula per DPS 3.114

20 grams	metallic sodium
100 grams	napthalene
1 liter	diethyleneglycol dimethylether

PROCEDURE: (CONTINUED)

b.	40 grams	metallic sodium
	200 grams	napthalene
	1 liter	diethyleneglycol dimethylether
c.	40 grams	metallic sodium
	200 grams	napthalene
	1 liter	tetrahydrofuran
d.	40 grams	metallic sodium
	300 grams	biphenyl
	1 liter	tetrahydrofuran

The solutions were prepared in the laboratory per DPS 3.114 and used within two weeks.

RESULTS:

Effect of Different Bondizing Formulations	Table 1
Coated Wires	Table 2
Effects of Varying Bondizing Time	Table 3
Effect of Ultraviolet Light on the Bondized Surface	Table 4

SIGNIFICANCE OF DATA:

The specially treated Surbond wire of Surprenant Mfg. Co., yielded significantly higher shear strength values than "inhouse" bondized TFE wires of the same diameter. Hitemps' Polymer Coat and Adbond increased adhesion slightly.

Of the seven solutions tested, neither type nor concentration has any significant effect on adhesion. As long as the solution is protected from atmospheric moisture in a closed container, it will remain effective for at least three months. However, when the solution is exposed to moisture an inactive layer forms on the surface. This layer forms a coating on the wire as it enters the bondizing solution and results in incomplete or spotty bondizing. This layer can be dispersed if the solution is agitated before use.

The duration of immersion in the bondizing solution is not critical. There are, however, variations in the color of the bondized Teflon insulation as related to the immersion time. The wires immersed for two seconds were slightly lighter in color than the wires immersed for two minutes; but there was no appreciable difference in the shear strength values.

SIGNIFICANCE OF DATA: (CONTINUED)

Results indicate that ultraviolet light has a severe degrading effect on bondized TFE insulation. Similar degradation will undoubtly occur on the bondized FEP. After exposure to ultra violet light the transitional carbonaceous surface film disappears and results in loss of adhesion. This deleterious effect can be avoided by completely excluding light until used. Red cellophane does give partial protection, however a completely opaque covering is desirable.

REFERENCES:

Case Sheets 30956, 40078, 36834 and 36847
TR Book 00067, pages 1 to 16
DPS 3.114
EWO 72805
JWO 7001
SO 3000-1071

C. T. McMurray

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Development, Components and Materials Section
Electrical Branch
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Approved: H. K. Lauer 7/31/64
H. K. Lauer, Section Chief
Development, Components & Materials
Electrical Branch
Materials Research & Production
Methods Department

CTMcM:ev

MATERIALS INDEX

1. Wire, TFE (polytetrafluoroethylene) insulated, single conductor, conforming to specification control drawing 7869679-B22.

2. Wire, FEP (fluorinated ethylenepropylene) insulated, single conductor, shielded conforming to specification control drawing 7869679-B20-ISJ.

3. Wire TFE insulated, single conductor. The insulation of this wire is surface treated by graft copolymerization. This process is performed by the manufacturer to improve adhesion. Surbond Type EE, 22 gage, catalog number Rte 1934 Nic (SB) and Surbond Type E, 20 gage, catalog number Wte 1932 Nic (SB).

   Surprenant Mfg. Company,
   Los Angeles, California

4. Bondaid (DPM 3393) a Teflon etching solution containing a sodium-napthalene complex.

   U. S. Shombam Company
   Los Angeles, California

5. Tetra-Etch (DPM 3128) a Teflon etching solution containing a sodium napthalene complex.

   W. L. Gore and Associates, Inc.,
   Los Angeles, California

6. Polymer Coat and Adbond. Two solutions used in sequence for the purpose of promoting adhesion to Teflon wire.

   Hitemp Wires Company
   Monrovia, California

7. Hysol 2039 (DPM 3084-1) (MRD-9709466 Type II) a clear, transparent, 100% solids, low viscosity epoxy potting compound. Cured by the addition of 30 parts by weight of Hysol 3561 (DPM 3084-2) to 100 parts of resin.

TABLE 1

THE EFFECT OF DIFFERENT BONDIZING FORMULATIONS

GROUP NUMBER	BONDIZING SOLUTION	PULL OUT SHEAR STRENGTH* TFE	FEP
1	None (controls)	11.6	14.1
2	Bondaid	55.4	28.5
3	Tetra-Etch	55.9	28.2
4	Formula a	60.5	27.6
5	Old DACo Production Solution	62.4	28.8
6	Formula b	54.3	28.9
7	Formula c	55.4	38.6
8	Formula d	60.5	29.8

* Averages of five specimens in lbs/sq.in.

TABLE 2

WIRES COATED FOR ADHESION (NON BONDIZED)

GROUP NUMBER	COATING TYPE	PULL OUT SHEAR STRENGTH* TFE	FEP
9	Treated with Polymer Coat and Adbond	19.8	23.2
10a	Surbond Type E (.062")	76.2	**
10b	Surbond Type EE (.060")	92.7	**

* Averages of five specimens in lbs./sq.in.

** This coated wire not available with FEP-insulation.

GORE

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TWX: 510/666-0825 · CABLE: GOREASSOC.

Technical Data: Tetra-Etch Teflon Etchant

1. General

Tetra-Etch® prepares fluorocarbon surfaces, such as "Teflon*", for potting, bonding, and marking. It is extremely simple to use with standard adhesives or potting compounds. The etchant is suitable for use in the laboratory or on the production line, and is among the safest materials made for this purpose.

2. Composition

Tetra-Etch is manufactured as a super-saturated solution of a sodium aryl compound in highly polar solvents. No free metallic sodium is present. Precise composition is considered proprietary information. The solvents used are rated non-toxic in reasonable exposures.

3. Properties

Color: Dark greenish-black
Specific Gravity: approximately .97
Boiling Point: approximately 85° C.
Flash Point: 33° F.
Auto Ignition Point: >745° C.
Solids Content: approximately 32%
Titration: (Butyl Alcohol) >0.5 gram equivalents of active sodium compound per liter. End point is disappearance of dark color.
Nonpyrophoric: will not ignite on contact with water

4. Use Reaction

The active sodium in Tetra-Etch reacts with the surface molecules of Teflon, removing fluorine atoms and leaving free radicals. These free radicals react with O_2, H_2O, and each other to form a miscellaneous group of compounds attached to the surface of the Teflon. Typical compounds formed may include ethylenic, acetylenic, hydroxyl, carbonyl, ketonic, epoxy, and other chemical groups. Many of these are reactive with epoxy and elastomeric adhesives or potting compounds, forming true chemical bonds with them. In addition, physical bonds are formed due to adhesive forces and to mechanical attachment on surface irregularities.

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*Trademark

The reactive film formed by Tetra-Etch is decomposed by ultra-violet and only a few weeks exposure to summer sunlight will destroy it. However, etched articles can be stored for long periods (years) in subdued daylight without losing their capability to form adhesive bonds.

The strength of an adhesive bond with the resultant etched Teflon surface will vary according to adhesives used and surface geometry. A typical epoxy adhesive will produce from 17 lbs./inch peel strength on smooth Teflon, to approximately 35 lbs./inch on the corrugated surface of Gore Multi-Tet ribbon cable.

The Tetra-Etch reaction is self-limiting as the film formed on the Teflon surface retards further etching. This makes precise timing of the exposure unnecessary.

Only a few seconds is required to obtain a good etch with Teflon. Prolonged exposure will darken the color and deepen the etch but offers no significant improvement in adhesion.

5. Other Reactions

Tetra-Etch may be used on any highly halogenated polymer with results varying according to the proportion of halogen present. FEP Teflon, polychlorotrifluoroethylene (Kel-F), polyvinylidene fluoride (Kynar), are etched to give reasonably good bonds with adhesives. These materials require a longer time than does TFE Teflon to acquire a good etch.

6. Procedures

1. Spot etching: Dip ends of Teflon tubing, tape or coated wire into the bottle of Tetra-Etch, or swab solution onto the surface with a dry applicator. After the dark color of the etchant disappears, clear off the residue with a damp cloth or flush off with acetone or methanol.

2. Batch treatment: Place Tetra-Etch in a container swept with dry nitrogen. Next, immerse the coils of wire, racks, or baskets of parts for about one minute. Then rinse treated parts of material in warm water containing a detergent followed by a final rinse in acetone, methanol, or naphtha gas.

3. Continuous lines: Place Tetra-Etch in a container swept with dry nitrogen. Run the wire or film through the solution, then through warm water containing a detergent. Traces of residue can be removed with acetone, methanol, or naphtha gas. Rates of 50 to 60 feet per minute are possible on continuous dip lines.

7. Safety Precautions

1. Tetra-Etch is flammable and should not be exposed to open flames.

2. Caustic soda is formed when the sodium reacts with water, therefore, any etchant spilled on the skin should be immediately flushed off with water. Rubber gloves are advisable. Safety glasses are recommended.

3. The solvents used are rated non-toxic in reasonable exposures. Ventilation should be provided sufficient to keep fumes below an objectionable level.

4. Tetra-Etch is toxic if taken internally, chiefly because of the caustic soda formed on reaction with moisture. Give milk or water, do not induce vomiting; obtain medical aid.

5. Since Tetra-Etch is nonpyrophoric and since it is mostly water soluble, left over or spent solution can be poured down the sink.

8. <u>Use Life</u>

Tetra-Etch will react with atmospheric humidity. In situations where prolonged exposure is necessary, a flow of dry nitrogen across the surface of the etchant will prevent premature depletion.

Tetra-Etch may be used for spot etching of wire by simply dipping the wire into the bottle. Reacted solution clings to the Teflon and is thus removed from the bath.

9. <u>Storage</u>

Exposure to heat has a pronounced effect on shelf life. For maximum life, refrigerated storage (50^{o} F. or less) is required.

Tetra-Etch is guaranteed for six months in unopened containers stored upright under refrigeration. Expiration dates are stamped on each container.

Etchant Makes Teflon Adhesionable for Bonding and Potting Applications

Chemical reaction with fluorocarbon resins produces a carbonaceous surface film which serves as a bonding medium in adhesive or potting applications

Cementing Teflon to itself or other materials has proved one of the more trying problems facing users of this versatile material. The polymer's inert chemical nature, its extreme anti-stick characteristics and the difficulties encountered in heat sealing it to itself have often combined to restrict and even discourage its use in many applications.

One of the knottier problems met with is to pot Teflon-covered wires so as to make them permanently watertight and highly resistant to shock impact. A new procedure developed by Kollmorgen Optical Co., Northampton, Mass., makes use of an active form of metallic sodium in solution to facilitate adhesion in attaching and potting plug assemblies to Multi-Tet ribbon cable.

Produces Surface Film

The etching solution, known as Tetra-Etch and manufactured by W. L. Gore and Associates, Newark, Del., reacts with fluorocarbon resins to produce a carbonaceous surface film which serves as a medium for tightly bonding most adhesive or potting compounds to the Teflon. While the Kollmorgen application is specifically a potting application, the same approach can be applied to all kinds of adhesive bonding applications as well. Here's how it works:

First, the flat multiple-conductor ribbon cable is prepared by slitting the homogeneous Teflon webbing between conductors, then stripping insulation from the conductor ends. For highest production, special cutting tools with multiple blades are available, or can be easily made.

The next step is to clean and dry all wires. The Teflon surface must be free of moisture and foreign matter. Then, cable ends are immersed in the etchant for about five seconds.

During the etching process, the color of the wires changes to a light brown. As a final preparatory step, acetone is employed to wipe all surfaces.

After wire ends and solder pots have been tinned and all leads are soldered into the plug, surfaces are wiped clean with methyl ethyl ketone (MEK) to rid them of possible contaminants such as grease, oils and sealants.

The assembly is now ready for priming. A thin coat of 3M Scotchcast Resin XR-5001 is brushed onto all surfaces and allowed to dry for several minutes. Then, in preparation for potting, the unit is placed in a mechanical fixture to assure rigid clamping during the resin cure. At the same time, Duct Seal is applied to all areas where uncured resin might leak out.

Finally, the assembly is potted with either Scotchcast No. 212 or No. 1120, depending upon service requirements. Curing cycles vary with the unit's size. For large masses of resin, an eight- to 16-hour cure at room temperature is followed by two to four hours under heat lamps placed about two feet from the resin surface. For thin sections, such as quick-disconnect plugs, a four- to six-hour cure with a heat lamp two feet from the resin has been found sufficient.

By HOWARD W. ARNOLD
Production Manager
W. L. Gore & Associates, Inc.
Newark, Delaware

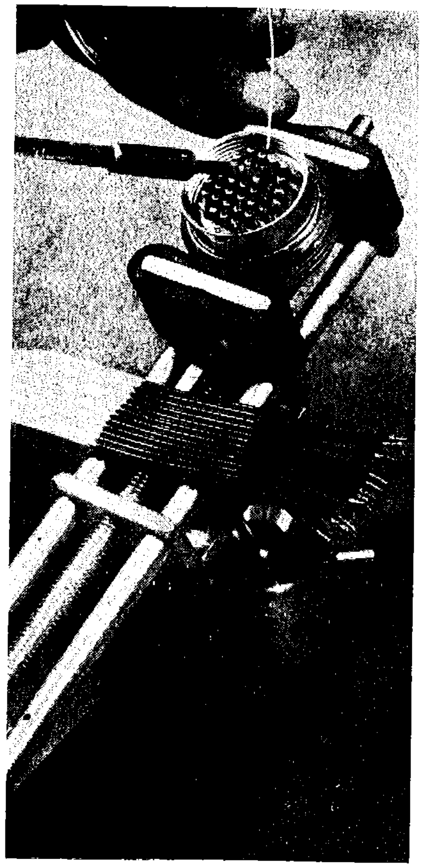
Exhaustive tests have shown that the resulting assembly withstands very rough treatment while maintaining a tight seal. As a practical demonstration of the unit's integrity, Kollmorgen engineers first closed off the front of the plug with Duct Seal: the entire assembly was then submerged in two feet of water, with only the extreme opposite end of the cable extending above the surface.

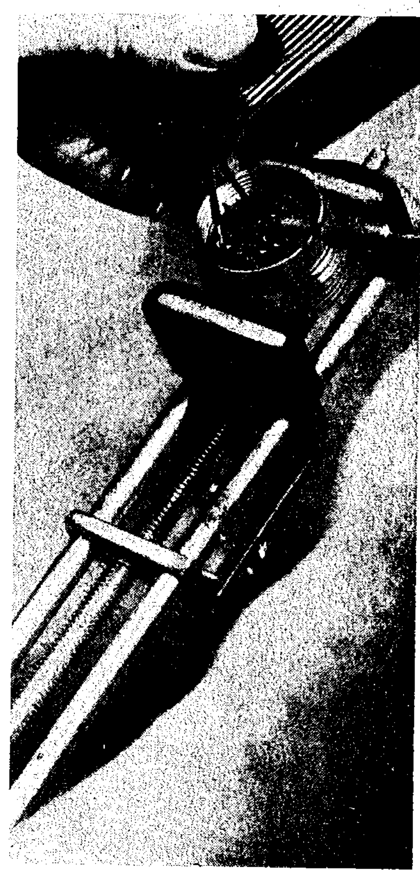
Testing Soaked Unit

After the unit had been soaked for a full week, all adjacent wires were tested with a 500-v. dc megger. No measurable leakage was detected from any wire to the water, or from wire to wire.

Next, the potted assembly was subjected to considerable mechanical abuse. Wires and entire cables were twisted, bent, and pulled in every direction. The plug then went back into the bath for another week, this time in a borax-and-water solution to decrease surface tension and increase conductivity. Again, results of the megger test were negative—there was no measurable leakage.

Although all Kollmorgen's work has been on TFE fluorocarbon resin, tests have shown that Tetra-Etch is effective on the more recent type 100 FEP Teflon as well. On Type 100, reaction time is usually a bit longer, but this tendency can be suppressed by heating the etchant to 50 to 60°C., or by heating the Teflon to about 100°C. Variations in tempera-





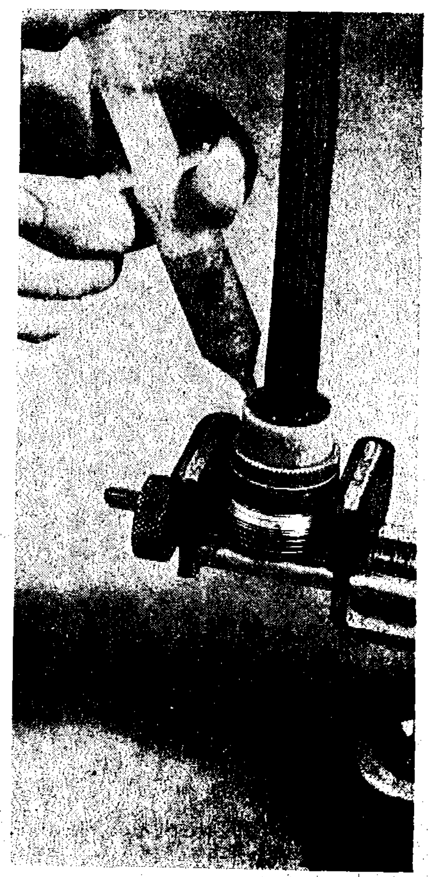
After rag-wiping to remove foreign matter, prepared ribbon cables are immersed in the etchant, top photo. The residue is removed with water and all cable surfaces are then wiped with acetone.

Pots are carefully tinned preparatory to actual soldering. Note wide spacing of conductors in cable. This can be varied to provide control of capacitance, mutual inductance and other properties, top center.

When the remaining leads have been soldered into the plug, top right, all surfaces are carefully wiped clean with methyl ethyl ketone to again make sure that oils, grease and other contaminants have been removed.

Primer is brushed on sparingly, photo right, and then allowed to dry for several minutes. Duct Seal is applied to all areas where uncured resin might possibly leak out of the pot which is to contain it.

Injection of the potting compound is shown in the photo on the far right. The mechanical fixture in which the unit is held assures rigid clamping during the subsequent resin cure under the heat lamps.



ture also dictate TFE resin's etching rate; this is an important point in setting up standard procedures for adhesive-bonding or potting.

There is yet another variable to remember: strength of the solution declines upon prolonged exposure to air. The reason for this is that the sodium in Tetra-Etch reacts with atmospheric oxygen, moisture and carbon dioxide to form sodium hydroxide. However, a two-ounce bottle can be left open to the air for over an hour without appreciable loss of etching ability.

Two ounces of the etchant will generally treat one to three square feet of surface. Variables here are atmospheric conditions, and how well the compound's potency is conserved.

Ultimate Strength

Peel strength of the adhesive bond of sodium-treated Teflon is ultimately limited to the adhesion of the carbonaceous film to the polymer. Peel-strength values range from 15 to 35 pounds per inch for cemented Teflon films, depending on film thickness, peeling rate, smoothness of surface, and adhesive system. For most applications, bonds of good durability are made with a moderately flexible epoxy composition.

With elastomeric adhesives (and all adhesives to a lesser degree) a considerable improvement in bond strength is obtained if the etched Teflon is first treated with an epoxy primer. The primers are made with a 10-15 per cent solution of catalyzed epoxy in toluene. The solution is dipped or brushed over the etched Teflon surface, then cured by heating. In a typical test using Dow-Corning 271 silicone adhesive, the maximum weight supported at 90° to the surface without peeling off the film was more than doubled with a primed surface.

While smaller items can be immersed in a bottle of Tetra-Etch for treatment, large-scale dip treatments require a blanket of nitrogen to prolong the etchant's life. Medium-sized Teflon objects can be etched by placing them in a polyethylene bag, pressing out the excess air, pouring a small amount of Tetra-Etch into the bag and manipulating the bag to bring the etchant into contact with all parts of the Teflon surface.

Tetra-Etch should be kept away from open flames because the liquid contained in the etchant is inflammable (although Tetra-Etch is non-pyrophoric). The small percentage of metallic sodium in the fluid reacts with moisture to form sodium hydroxide; thus, any fluid that is spilled on the skin should be flushed off immediately with water.

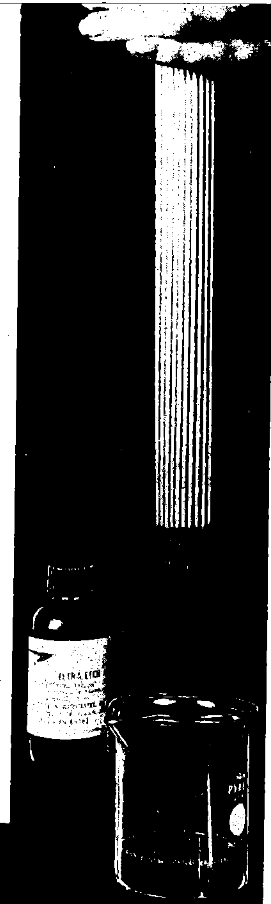
While the liquid is a powerful solvent for most plastics and lacquers (and nearly all elastomers are swollen and softened by it), glass, polyethylene and all metals are never affected.

Summing Up

In summary, Tetra-Etch provides a simple solution for what had been a baffling problem—bonding Teflon. Any highly halogenated polymer is etched by the metallic sodium composition, including chlorotrifluoroethylene resin (Kel-F, Fluorothene), the hexafluoropropylene copolymer with tetrafluoroethylene (Teflon 100 FEP resin), and Viton fluorinated rubber.

About the Author . . .

HOWARD W. ARNOLD, production manager of W. L. Gore & Associates, Inc., attended the University of Akron and the University of Delaware. Before joining Gore, Mr. Arnold was a production supervisor for Goodyear Aircraft Corp., plant manager for General Development Corp., and plant material engineer at the Chrysler Delaware Assembly Plant.



TETRA-ETCH® TO PREPARE TEFLON FOR BONDING

TETRA-ETCH is a fluid for treating the surface of Teflon to make it bondable. The TETRA-ETCH fluid contains a sodium compound that reacts with any halogenated polymer to form a reactive film on the treated surface. Nearly any adhesive will adhere to this treated surface.

The etching is effected by merely pouring TETRA-ETCH on the surface to be treated or by dipping into the etchant.

Peel strength of bonds will vary with surface geometry and the adhesive used. A typical epoxy adhesive will produce from 17 lbs. per inch on smooth Teflon to about 35 lbs. per inch on the corrugated surface of Teflon MULTI-TET (multiple-conductor) ribbon cable. Treatment time ranges from a few seconds to a full minute, depending on temperature and strength of the solution. With FEP Teflon and some other materials, the reaction is a little slower. This can be offset by heating the Teflon to about 100° C.

Exposure to air effects the compound's strength since the sodium compound in TETRA-ETCH reacts with oxygen, water, and carbon dioxide. However, a 2 oz. bottle may be left open to the air for an hour or so without appreciable loss of etching ability. Heat will also degrade the etchant, as shown in the accompanying chart. TETRA-ETCH loses its normal dark color as its exhaustion approaches. The etching reaction in itself has no effect on strength, because reacted solution clings to the Teflon and is removed from the bath.

Adhesives: All types of adhesives bond well to the carbonaceous film which TETRA-ETCH forms on Teflon. This includes epoxy adhesive and potting compounds, silicone rubbers, phenolformaldehyde and similar compounds, elastomeric adhesives and potting compounds, and a wide variety of solvent-cast and polymerizable-resin adhesives. The following are typical recommendations:

For maximum bond strength and resistance to vibration, use a medium-flexibility epoxy adhesive such as C. H. Biggs, Helix Bonding Agent R-363, Emerson & Cumings, Eccobond 45, or Rubber and Asbestos Corp., G-379. For a pressure-sensitive adhesive satisfactory up to 390° F. (200° C.), use Dow-Corning C271 silicone or similar materials.

Spot Etching: Dip ends of Teflon tubing, tape or coated wire into the TETRA-ETCH bottle or pour on surface and quickly spread with a metal or polyethylene strip. After a short time or when the compound's dark color disappears, clean off the residue with a damp cloth or flush off with water. The etched area should be light brown. Further cleaning can be done with acetone, methanol, or naphtha gasoline.

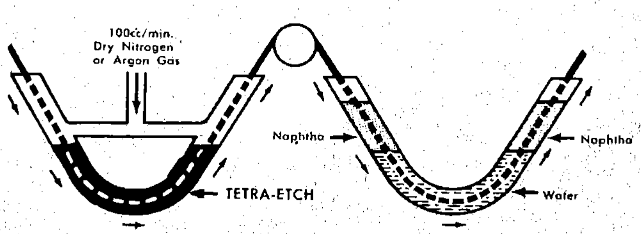
TETRA-ETCH® TO PREPARE TEFLON FOR BONDING

Batch Treatment: Place TETRA-ETCH in a container swept with dry nitrogen or argon. Next, immerse the coils of wire, racks or baskets of parts for about one minute. Then rinse treated parts of materials in warm water containing a detergent and make a final rinse in acetone, methanol, or naphtha.

Continuous Lines: Place TETRA-ETCH in a container swept with dry nitrogen or argon. Now run the wire or film through the solution and then through warm water containing a detergent. Traces of residue can be removed with acetone or methanol or vaporized by postheating for an hour at 250 - 275° F. in an oven. Rates of 50 to 100 fpm are possible on continuous-dip lines.

Gore Associates is equipped to do spot, batch and continuous etching on a job-lot or a production basis. Since continuous etching of Teflon insulated wire and ribbon cable can be done in conjunction with Gore's normal manufacture process, etched wire and cable can be furnished at little additional cost.

CONTINUOUS ETCHING SYSTEM

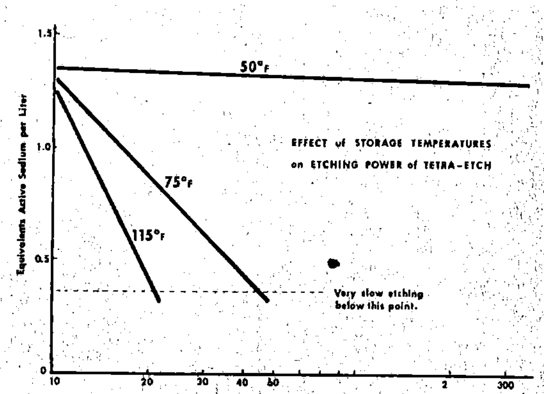


Potting: When treated with TETRA-ETCH, Teflon insulated wires can be potted into electrical connectors. A water-tight assembly can be achieved which is highly resistant to shock impact. (For details of a typical application, send for Gore article No. T-E 1001.)

TETRA-ETCH treatment results in a film on the surface of Teflon to which most marking inks will adhere tightly. Many of these inks dry quickly and provide durable printing or identification marks. Speeds of automated wire marking equipment can be greatly increased by use of etched Teflon wire and fast-drying ink.

Composition: TETRA-ETCH is manufactured as a super-saturated mixture of a sodium aryl compound in highly polar solvents. As no free metallic sodium is present, TETRA-ETCH is nonpyrophoric; will not ignite when exposed to air or poured into water. This is an exclusive and important feature which eliminates a major fire hazard. The solution is dark greenish-black, with a specific gravity of approximately .97 and a vapor pressure of approximately 50 mm Hg at 20° C. Its flash point is approximately 33° F; the auto-ignition temperature is greater than 745° C.

Precautions: TETRA-ETCH is inflammable and should be kept away from open flames. Caustic soda is formed when the sodium compound reacts with moisture; therefore, any solution that gets on the skin should be flushed off immediately with water. Rubber gloves are advisable and safety glasses should be worn.
Provide good ventilation to prevent prolonged breathing of fumes from the solution.
Disposal of spent TETRA-ETCH is simple; pour it down the drain.



Shipping and Storage: Polyethylene bottles used to package TETRA-ETCH are hermetically sealed inside tin cans. Shelf life is guaranteed for six (6) months when stored below 50° F. Refrigeration is not required for immediate use (within a week). Air shipments are recommended to insure maximum strength. TETRA-ETCH orders are filled from fresh stock, usually on the same day the order is received.

W. L. GORE & ASSOCIATES, INC.
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W. L. GORE & ASSOCIATES, INC.

555 PAPER MILL ROAD, NEWARK, DELAWARE

TWX 302 737 1060

COMMENTS ON BONDABILITY AND SHELF LIFE OF FLUOROCARBONS AFTER TREATMENT WITH TETRA-ETCH

BONDABILITY WITH TFE

The active sodium compound in TETRA-ETCH reacts with the surface molecules of Teflon, removing fluorine atoms and leaving free radicals. These free radicals react promptly with oxygen, water, or ingredients of the etchant or cleaning baths to form a layer of chemical groups attached to the surface of the Teflon. The groups are likely to be ethylenic, acetylenic, hydroxyl, carbonyl, ketonic, epoxy, etc. This layer of attached groups provides a bondable surface, which will adhere to most adhesives and potting compounds.

The reactive film is brownish in color, and is frequently used as evidence of successful etching. Bondability is indicated by the change of color, and not by the hue or darkness, which is really an indication of the composition and thickness of the film layer. The buildup and color of this layer are influenced by the length of exposure to the etchant, the cleaning baths or to air; the strength of the etchant, the nature of the cleaning agents, etc.

The thickness of the reactive layer plays a role in bond strength with adhesives or potting compounds. Generally, a thick layer is not as strong as a thin one, and is more likely to be separated from the base polymer. This has been demonstrated by over-etching wires for extended periods, causing a heavy layer to form. After aging, it will be found in some cases that the film layer may be loosened and slid from the wire. Interestingly, the wire may be potted after removal of the film and will still yield a fairly good bond.

OTHER FLUOROCARBONS

FEP Teflon reacts more slowly, and the film buildup is less obvious. For this reason, longer exposure to the etchant is recommended. (60 seconds should produce a good etch with FEP.) The reaction may be speeded up by pre-heating the wire to 100^{o}C, and nitrogen blanketing is more important. The film layer on FEP is seldon as dark in appearance as that obtained with TFE. Kel-F is even less reactive.

HANDLING AND STORAGE

Storage of etched wires will result in a gradual lightening of the color, as the film layer breaks down into more stable groups. Ultra violet radiation destroys the attachment of the etched film to the substrate, so the etched product should be protected from UV light. Although the etched surfaces lose some of their distinctive color, experimental evidence from samples several years old indicates there is no loss, and may be a slight improvement, in bond strength.

The etched film is rather tender at first and can be easily scraped off the Teflon. The aging reactions, particularly in the first few hours, harden the film and make it more resistant to mechanical damage. The use of an epoxy primer on freshly etched surfaces provides a mechanical protection for the bonding treatment.

SUMMARY

TETRA-ETCH produces a reactive film which is chemically bonded to the surface of the fluorocarbon base material. A change of color occurs as an immediate result of etching, indicating that the film layer has been formed. This film may in turn be bonded to a variety of adhesive materials. An extremely dark film indicates a heavy buildup of compounds on the surface of the fluorocarbon, which may lead to separation of the film layer itself.

Once etched, bond strength which can be achieved with some particular adhesive remains relatively constant over several years at least, regardless of changes in the color of the surface film.

Etched materials should be handled as little as possible, especially in the first few hours after etching, to avoid mechanical damage to the film layer. They should be stored in a place protected from UV light.

April , 1965

TABLE 3

EFFECT OF VARYING TIME IN BONDIZING SOLUTION

GROUP NUMBER	IMMERSION TIME IN FORMULA a	PULL OUT SHEAR STRENGTH* TFE	FEP
11	2 seconds	54.9	30.2
12	10 seconds	54.3	29.0
13	30 seconds	60.5	27.5
14	120 seconds	53.4	29.5

NOTE: The wires bondized for 2 seconds were lighter in color than the wires bondized for longer periods.

TABLE 4

EFFECT OF ULTRAVIOLET LIGHT ON BONDIZED WIRES

GROUP NUMBER	TREATMENT OF WIRES AFTER BONDIZING 30 SECONDS IN FORMULA I	PULL OUT SHEAR STRENGTH* TFE ONLY
15	Total Darkness	40.6
16	Placed 2 feet from the high intensity Carbon Arc of the Weatherometer**	13.6
17	Wires were wraped with two layers of red cellophane *** and placed in Weatherometer with group 16 for 16 hours	32.4
18	Placed 1 foot from a "sun lamp" for 16 hours	11.5

* Averages of five specimens in lbs/sq.in.

** Weatherometer, Atlas Model XW. Without water spray.

*** DPM 720

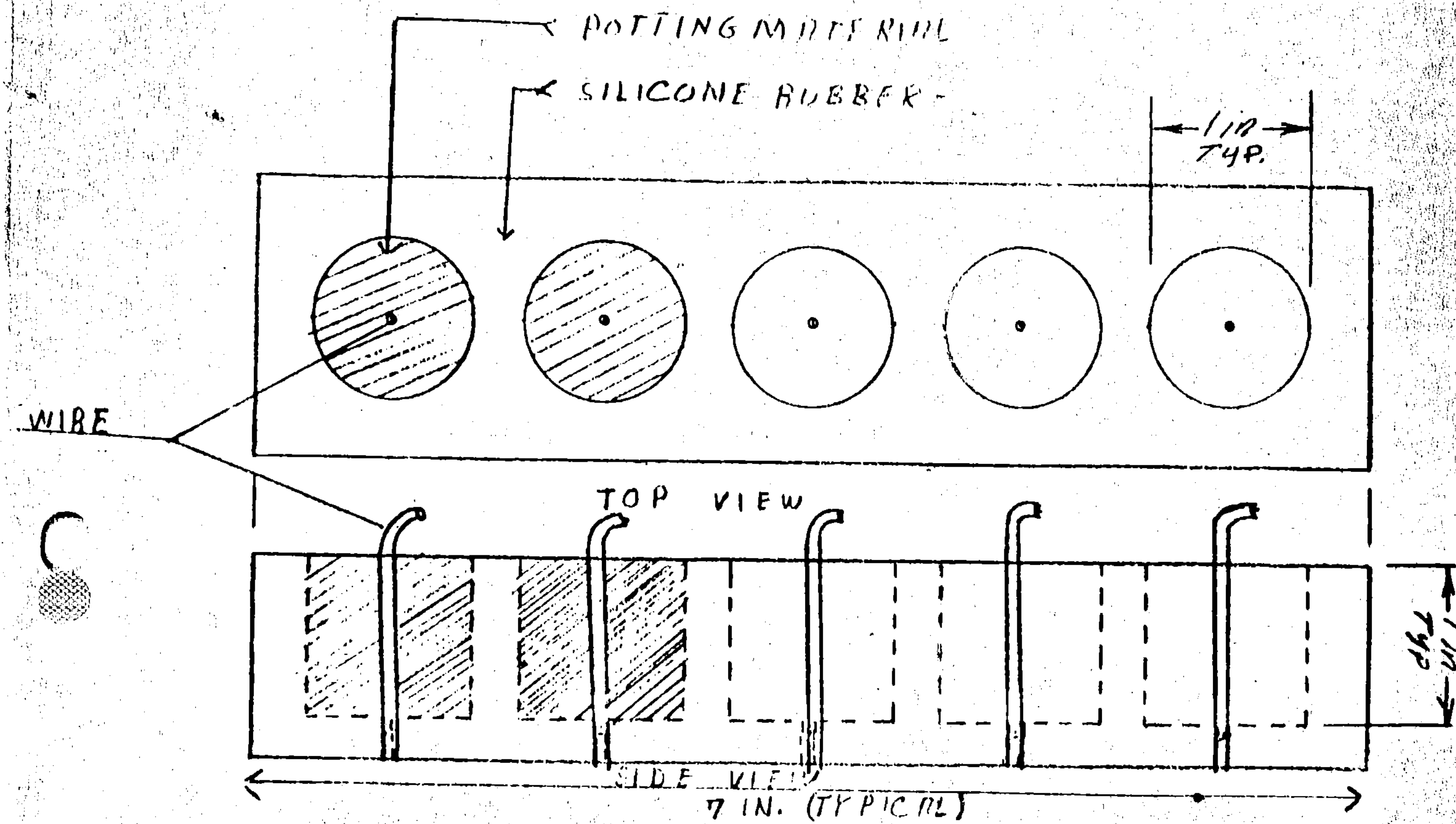

Figure I
Mold for Potting Wires